A MICROWAVE ANALOGUE FOR X-RAY DIFFRACTION PART I. EFFECT OF THE CRYSTALLITE SIZE

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ABSTRACT. Diffraction of X-rays by a crystal is analogous to that of microwaves by a three dimensional array of scatterers when the distance of one scatterer from another is of the same order of magnitude as the wave length of the microwaves. Thus all phenomena connected with the diffraction of X-rays by crystals are expected to be obtained due to the scattering of microwaves by a three dimensional arrangement of scatterers. To verify this, metallic scatterers in the form of small cylinders have been arranged to form the model of a crystal having tetragonal lattice parameter of $a = 3.2$ cm and $c = 4.8$ cm. A lattice of 900 such unit cells have been irradiated by microwaves of wavelength 3.2 cms. Bragg's law has been found to be valid for the (100) and (110) planes of this crystal model. The intensity distribution curves around these two reflection maxima have been studied for 10, 7 and 4 planes in the *b*-direction. The half intensity widths have been compared with the formula due to Scherrer. Scherrer formula has been found to agree fairly well with experimental data.

INTRODUCTION

As is well known, all the phenomena concerning diffraction of X-rays by crystals have been explained on the basis of extremely short wavelength electromagnetic waves being scattered by atoms placed at the lattice points of a three dimensional periodic structure. The scattered electromagnetic waves interfere with each other and in the directions they reinforce each other there is obtained a diffraction maximum. The pattern of the diffraction maxima depends on the arrangement of the atoms in the crystal. Thus a study of the diffraction pattern of a crystal reveals the arrangement of atoms in it.

If a lattice is constructed with metal or dielectric scatterers at the lattice points, the lattice constants being of the order of centimeters, its behaviour towards centimetre wavelength microwaves should be exactly similar to that of a crystal to X-rays. Recently Allen (1955) described the verification of Bragg's law for 12-cm wave length microwaves and a model cubic structure of lattice constant 16 cm having metal discs mounted on wooden rods. The model was eight planes long, 4 planes high and two planes deep and so consisted of sixty-four unit cells only. Allen obtained diffraction maxima in the first order as well as in the second order for the (100) planes only. Since this is the first experiment of its kind, it has been considered worth while to repeat the experiment for another crystallographic system, viz the tetragonal class with larger number of unit cells so that a crystal could be simulated more realistically. It has also been decided to study the effect of the crystal size on the width of the intensity distribution curves.

EXPERIMENTAL

A lattice containing nine hundred unit cells has been constructed with brass scatterers at the lattice points. The brass scatterers were cylinders having 1 cm diameter and 0.5 cm height and provided with a small hole in the middle for passing plastic threads through them. The plastic threads without scatterers were tested for their scattering power and were observed to have none or at least very little. This lattice was supported in a wooden structure provided with arrangements for quick rearrangement of the scatterers. The lattice constants in this case were $a = 3.2$ cm, $b = 3.2$ cm and $c = 4.8$ cm

The lattice was irradiated by a microwave horn and the diffraction pattern about the lattice was measured by another microwave horn Both these horns could be rotated about a common vertical axis of the lattice The microwave signal was generated by a klystron (723A/B) and was fed to the radiating horn The second microwave horn, acting as a receiving antenna, picked up the diffracted signal and fed into a crystal detector The klystron was square-wave modulated and the video square wave at the output of the detector was measured by an amplifier The diffraction pattern was obtained by recording the output of the amplifier at various settings of the receiving horn for a pre-determined position of the transmitting horn. The scattering due to the wooden frame and the walls etc. of the room was first of all studied to determine the zero level of the signal received by the detector. This was made very low compared to the signal from the three dimensional array The polar diagramme of the radiating horn was studied to find out the effect of instrumental broadening on the intensity distribution curves.

RESULTS AND DISCUSSIONS

The incident beam of microwaves was normal to the (001) direction and readings were taken only for the (100) and (110) planes. A diffraction maximum was obtained for both the incident as well as the diffracted beams making an angle of 30° with the planes parallel to the X-axis This corresponds to the Bragg angle for the (100) plane of the lattice under investigation Similarly, a diffraction maximum was again observed at 44° corresponding to the Bragg angle for the (110) planes. Thus Bragg law was found to be satisfied at least in these two cases.

Next the effect of particle size was investigated. The intensity *vs* angle curves in the vicinity of the 44° and 30° scattering maxima for 10, 7 and 4 planes in the b-direction were drawn and their half intensity angular widths were determined after eliminating the instrumental broadening The results are shown in Table I and in Figs. 1 and 2.

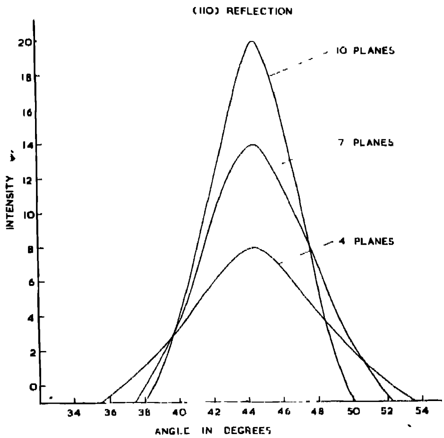

Fig. 1

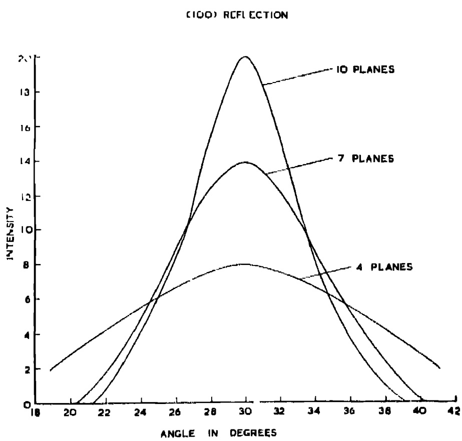

Fig. 2

The measured half intensity widths have been compared with those calculated from the well-known Scherrer formula (1918). While calculating the half intensity width

$$\beta = \frac{\lambda}{t \cos \theta}$$

where λ is the wavelength, θ the Bragg angle and t the crystallite size, t has been taken to be the thickness of the crystallite in the (hkl) direction. It is observed that the measured half intensity widths agree rather fairly well with the Scherrer formula. It is to be noted that all the planes normal to the (110) direction did not have the same number of scatterers in them. The end planes possessed very few scatterers It is clear that it is necessary to have the intensity curves for the planes free from overlap from scatterings due to other planes To achieve this, the lattice dimensions have been so taken that only two Bragg reflections, namely, at 30° and 44°30′ are possible for the given setting of the crystal model. Even then there has been certain overlap between these scatterings due to the two planes. The intensity distribution curves shown in figures 1 and 2 have been drawn after eliminating the effects due to such overlap. It must be noted that in calculating the half intensity widths the measurements of the parameters involved have been fairly accurate. The close agreement of the measured half intensity widths with Scherrer's formula is rather interesting.

ACKNOWLEDGMENT

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TABLE I

Plane (hkl)	Bragg angle θ degrees	Thickness of crystal in the (hkl) direction t cms.	Half intensity angular width calculated from Scherrer formula	Observed half intensity width β in degrees
		32.0	6°36′	7°
100	30°	22.4	9°48′	11°
		12.8	16°32′	17°
		44.8	5°44′	6°
110	14°30′	39.1	6°34′	7°
		34 5	7°28′	8°

REFERENCES

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